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(54) Purification Process for Bitumen Froth

(72) Shelfantook, William E. , Canada  
Hyndman, Alexander W. , Canada  
Hackman, Larry P. , Canada

(73) Alberta Energy Company Ltd. , Canada  
Canadian Occidental Petroleum Ltd. , Canada  
Esso Resources Canada Limited , Canada  
Gulf Canada Resources Limited , Canada  
Majesty (Her) the Queen in right of the Province of  
Alberta, as represented by the Minister of Energy and  
Natural Resources , Canada  
HBOG-Oil Sands Limited Partnership , Canada  
PanCanadian Petroleum Limited , Canada  
Petro-Canada Inc. , Canada

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Canadian Patent No. 1,293,465  
Granted: December 24, 1991

Les corrections suivantes sont faites en  
raison de l'article 8 de la *Loi sur les  
brevets* et le document doit être lu tel  
que corrigé.

In the Patent file and Patent grant:

The following corrections are made  
pursuant to section 8 of the *Patent Act*  
and the document should read as  
corrected.

1.

The following lines have been added to the top  
of page two of the claims:

"mixing the first underflow stream from the first settler  
in the second mixer with a second recycled overflow  
stream from the third settler, said second overflow  
stream being"

Agent certifiéur / Certifying Officer

November 29, 1999

Date



Industrie  
Canada

Industry  
Canada

(CIPO 25)

Canada

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1 "PURIFICATION PROCESS FOR BITUMEN FROTH"

2 ABSTRACT OF THE DISCLOSURE

3 Bitumen froth is treated in a circuit comprising a  
4 plurality of serially connected mixer and inclined plate  
5 settler units. A light hydrocarbon diluent moves  
6 countercurrently through the circuit. Thus, as the bitumen  
7 content of the stream being settled diminishes, the  
8 concentration of diluent in that stream increases.

FIELD OF THE INVENTION

This invention relates to a process for purifying bitumen froth, to thereby obtain a diluted bitumen stream of good enough quality to be fed to a downstream upgrading facility. By 'purifying' is meant that water and solids present in the froth are separated from the bitumen.

BACKGROUND OF THE INVENTION

The oil sands of the Fort McMurray region of Alberta are presently being exploited by two large commercial operations. The process practised in these operations involves four broad steps, namely:

- mining the oil sand;
- extracting the bitumen from the mined oil sand using a process known as the 'hot water process', to produce bitumen in the form of a froth contaminated with water and solids;
- purifying the froth to separate the water and solids from the bitumen; and
- upgrading the purified bitumen in a coking facility to produce products which are suitable for a conventional refinery.

The present invention has to do with the purifying step. However, in order to understand the problems solved by the invention, it is first necessary to review the steps of the hot water process and the conventional froth purification process.

As a beginning point, it needs to be understood that oil sands comprises relatively large quartz sand grains, each grain being encapsulated in a thin sheath of connate water. The water contains minute clay particles (referred to



1 as 'fines'). The bitumen is positioned in the interstices  
2 between the water-sheathed grains of sand.

3 In the first step of the hot water process, the  
4 mined oil sand is mixed in a rotating horizontal cylindrical  
5 drum (or 'tumbler') with hot water (80°C) and a small amount  
6 of NaOH (referred to as 'process aid'). Steam is sparged  
7 into the slurry at intervals along the length of the drum, to  
8 ensure that the exit temperature of the resultant slurry is  
9 about 80°C.

10 The drum is slightly inclined along its length, so  
11 that the mixture moves steadily therethrough. The retention  
12 time is about 4 minutes.

13 This tumbling step is referred to as  
14 'conditioning'. It involves heating of the bitumen and  
15 displacement, by water addition, of the bitumen away from the  
16 sand grains. Many of the released bitumen globules become  
17 aerated by forming films around air bubbles entrained in the  
18 tumbler slurry. Conditioning also involves reaction between  
19 the NaOH and bitumen to produce surfactants which facilitate  
20 the bitumen-release and subsequent flotation/settling steps.

21 On leaving the tumbler, the conditioned slurry is  
22 screened, to remove oversize rocks and lumps, and diluted  
23 with additional hot water. The resulting water/bitumen ratio  
24 is about 6:1.

25 The diluted slurry is then introduced into a large  
26 thickener-like vessel having a cylindrical upper portion and  
27 a conical lower portion. The vessel is referred to as the  
28 'primary separation vessel' or 'PSV'. Here the diluted  
29 slurry is retained for about 45 minutes under quiescent  
30 conditions. Under the influence of gravity, the sand grains  
31 sink, are concentrated in the conical portion and are

1 discharged as 'primary tailings' through a valve and line  
2 connected to the lower apex of the vessel. The bitumen  
3 globules, rendered buoyant by air attachment, rise to the  
4 surface of the PSV and form a froth. This froth is called  
5 'primary froth' and typically comprises:

6 66.4% by wt. bitumen

7 24.7% by wt. water

8 8.9% by wt. solids

9 The primary froth is skimmed off and recovered in a launder.  
10 In between the layer of sand tailings in the base of the  
11 vessel and the layer of froth at the top, there exists a  
12 watery slurry referred to as 'middlings'. The middlings  
13 contain fines and globules of bitumen which are  
14 insufficiently buoyant to reach the froth layer.

15 A stream of middlings is continuously withdrawn  
16 from the PSV. These middlings are treated in a series of  
17 sub-aerated flotation cells. In these cells, the middlings  
18 are vigorously aerated and agitated, with the result that  
19 contained bitumen is forced to float and form a dirty froth  
20 referred to as 'secondary froth'. This secondary froth  
21 typically comprises:

22 23.8% by wt. bitumen

23 58.7% by wt. water

24 17.5% by wt. solids.

25 To reduce the concentration of water and solids in  
26 the secondary froth, it may be retained in a settling tank to  
27 allow some of the contaminants to settle out. The 'cleaned'  
28 secondary froth typically comprises:

29 41.4% by wt. bitumen

30 46.2% by wt. water

31 12.4% by wt. solids.

1           The primary and secondary froths are then combined  
2 to provide the product of the hot water extraction process.  
3 The 'combined froth' typically comprises:

4           57.3% by wt. bitumen  
5           34.2% by wt. water  
6           8.4% by wt. solids.

7           This stream is too contaminated to be used as feed  
8 to the downstream upgrading circuit. This latter circuit  
9 requires a feed typically comprising:

10          99.0% by wt. bitumen  
11          -   % by wt. water  
12          1.0% by wt. solids.

13          So the combined froth product requires purification  
14 (or water and solids removal) before it can be fed to the  
15 upgrading circuit. Heretofore, this purification has been  
16 obtained by using what is referred to as 'two stage dilution  
17 centrifuging'. This operation involves:

- 18          1. Diluting the combined froth with naphtha.  
19             This is done to reduce hydrocarbon phase  
20             viscosity and increase the density  
21             difference between the hydrocarbon phase  
22             (bitumen dissolved in naphtha) and the  
23             water and solids phase (referred to jointly  
24             as 'sludge');  
25          2. Passing the diluted froth through a low-  
26             speed scroll centrifuge, to remove the  
27             coarse solids and some of the water as a  
28             cake , which is discarded; and

1                   3.    Passing the scroll centrifuge product  
2                    through a high-speed disc centrifuge to  
3                    remove fine solids and most of the balance  
4                    of the water.   The disc centrifuge product  
5                    typically analyzes at:

6                               59.4% by wt. bitumen

7                               37.5% by wt. naphtha

8                               4.5% by wt. water

9                               0.4% by wt. solids

10                   The naphtha diluent and any contained water is then  
11                   distilled out of the disc centrifuge product to produce the  
12                   purified bitumen product for advancing to the upgrading  
13                   process.

14                   The described dilution centrifuging process has  
15                   been used because it is capable of producing a bitumen  
16                   product of the desired quality.   But it is an operation that  
17                   is exceedingly expensive to maintain and operate due to the  
18                   erosive nature of the feed and the rotating character of the  
19                   centrifuges.   For example, in use, the flights of the scroll  
20                   centrifuges wear badly, even though they are formed of  
21                   ceramic, and the brittle ceramic flights commonly break and  
22                   put the machine out of balance.   In the case of the disc  
23                   centrifuges, their sludge discharge nozzles are subject to  
24                   rapid wear and the separation interface between product and  
25                   reject in the stack of discs can easily be 'lost', with the  
26                   result that a significant amount of bitumen is lost with the  
27                   tailings.   In addition, a large number of the machines must  
28                   be used, with attendant consumption of very large amounts of  
29                   electrical energy.



1           Thus, there has long been a need for a viable  
2 alternative to the dilution centrifuging circuit for  
3 purifying bitumen froth.

4           The present invention involves a circuit of  
5 interconnected known devices, namely mixers and inclined  
6 plate settlers ('IPS').

7           An inclined plate settler comprises a stack of  
8 parallel, spaced apart, solid plates, inclined downwardly  
9 from the horizontal and mounted within a containing vessel.  
10 Each space between a pair of plates forms a discrete settling  
11 zone. The feed mixture to be separated is distributed into  
12 the spaces, at a point between their longitudinal ends. The  
13 light components of the mixture rise to the underside surface  
14 of the upper plate. These light components then travel up  
15 said underside surface and are collected and recovered at the  
16 upper ends of the plates. The heavy components of the  
17 mixture sink towards the uppermost surface of the lower plate  
18 and follow it downwardly, to be collected and recovered at  
19 the lower ends of the plates.

20           A mixer can take any of various forms - the present  
21 work involved simply a cylindrical container having a  
22 submerged driven impellor positioned therein.

#### 23           SUMMARY OF THE INVENTION

24           The present invention is based on the following  
25 experimentally determined observations:

- 26           - That bitumen froth is amenable to high quality  
27 separation in a first IPS, but in that first  
28 stage of separation only part of the bitumen  
29 in the feed reports as overhead product;

- 1                   - That the underflow from the first IPS, containing  
2                   a significant proportion of the bitumen in the  
3                   original feed, is not amenable to high quality  
4                   separation in a second IPS. It appears that the  
5                   first stage underflow contains stable emulsions  
6                   that will not readily resolve in the second IPS  
7                   or that much of the hydrocarbons that did not  
8                   report to the overflow in the first stage will  
9                   also not report to the overflow in the second  
10                  stage; and  
11                  - That if light hydrocarbon diluent (e.g. naphtha)  
12                  is mixed with the first stage underflow, then  
13                  this mixture is amenable to good quality  
14                  separation in the second IPS.

15                  Having conceived and tried the underlying  
16                  experimental work that resulted in these observations,  
17                  applicants conceived a purification circuit for bitumen froth  
18                  that would incorporate the following features:

- 19                  - the use of a plurality of serially connected  
20                  inclined plate settlers, with a subsequent  
21                  settler being fed the underflow from a  
22                  preceding settler;  
23                  - the addition of light hydrocarbon  
24                  diluent or solvent, in a progressively  
25                  richer concentration, to the bitumen-  
26                  containing stream moving through the  
27                  series of settlers, said bitumen-containing  
28                  stream becoming progressively leaner in  
29                  bitumen as it moves through the circuit; and

- 1           - the use of mixers before each settler to mix  
2           the added diluent with the bitumen.

3   A circuit or line consisting of three pairs of alternating mixers  
4   and settlers was tested. The overflow stream from the first  
5   settler provided the only bitumen product stream produced from  
6   the circuit. The bitumen/diluent overflow stream from the second  
7   settler was recycled to the first mixer to be combined with the  
8   froth feed. The low-bitumen/high-diluent overflow stream from  
9   the third settler was recycled to the second mixer. Thus more  
10   diluent was supplied to the relatively bitumen-lean underflow  
11   stream being supplied to the second mixer. And finally, fresh  
12   diluent was supplied to the third mixer to dissolve the small  
13   amount of bitumen in the underflow stream of the second settler.

14           When applied to typical combined bitumen froth this  
15   circuit demonstrated:

- 16           - that the bitumen product stream from the first  
17           IPS was of the same order of purity as that  
18           derived from a conventional dilution centrifuging  
19           circuit; and  
20           - that the recovery of bitumen by the test circuit  
21           was of the same order as that obtained by  
22           dilution centrifuging.

23           Stated otherwise, we have made the surprising discovery  
24   that a process using three mixing/IPS separation steps in  
25   series, combined with a counter flow of solvent, gives product  
26   of as good quality as that obtained from the centrifuge process  
27   (said quality being referred to as "upgrading quality"),  
28   together with comparable hydrocarbon recovery and a sludge  
29   tailings that is substantially hydrocarbon-free. And the

1 components of the present circuit are without moving parts  
2 (except for the pumps and impellers) and thus are  
3 characterized by comparatively low maintenance costs.

4 DESCRIPTION OF THE DRAWINGS

5 Figure 1 is a block diagram showing the steps of  
6 the process in accordance with the preferred embodiment; and

7 Figure 2 is a schematic showing the circuit of  
8 processing components or units and their pipe  
9 interconnections.

10 DESCRIPTION OF THE PREFERRED EMBODIMENT

11 The test work underlying the present invention was  
12 carried out in 3-stage mixer/IPS circuit. The invention will  
13 now be described with respect to that circuit, although it  
14 could also be conducted in 2, 4 or even more stages.

15 More particularly, combined bitumen froth was fed  
16 to a circuit A comprising: a first mixer 1; a first IPS 2; a  
17 second mixer 3; a second IPS 4; a third mixer 5; a third IPS  
18 6; and appropriate connecting lines.

19 The combined froth was introduced into and mixed in  
20 the first mixer 1 with a first recycled overhead stream from  
21 the second IPS 4. This first recycled overhead stream was  
22 depleted in bitumen but enriched in naphtha, relative to the  
23 combined froth feed.

24 The first mixer 1 comprised a cylindrical body 1a  
25 having a flat bottom 1b. An impellor 1c was positioned to  
26 stir the contents of the mixer.

The mixture from the first mixer 1 was fed to the inlet of the first IPS 2. The first IPS 2 was simply a box 2a having an inlet 2b, an overhead outlet 2c, and an underflow outlet 2d. The box contained a pair of inclined spaced-apart plates 2e.

The dimensions of the mixer and IPS units used are set forth in Table 1. The several mixers and IPS's in the circuit were identical to the described units.

TABLE I

Length of IPS -	5'
Spacing between plates -	1-1/2"
Dimensions of plates -	5' x 1'
Mixer vessel -	12" diameter
	12" to 16" of liquid in the vessel during operation
Type of impellor -	6" diameter marine propeller
Impellor rpm -	220 - 680
Separation of the bitumen, water, and solids, present in the mixture fed from the first mixer 1, took place in the first IPS 2. A first overhead product stream, which was the only bitumen-rich product from the circuit, was obtained. This stream was enriched in bitumen relative to the original froth feed. (The compositions of these streams are set forth in Table II below.)	

The underflow stream from the first IPS 2 was fed to the second mixer 3. Here it was mixed with a second recycled stream from the third IPS 6. This second recycled stream was very depleted in bitumen but relatively rich in naphtha.

1           The mixture from the second mixer 3 was fed to the  
2 inlet of the second IPS 4. Separation occurred therein and  
3 overflow and underflow streams were produced. The overflow  
4 stream was the stream recycled to the first mixer, as  
5 previously stated.

6           The second underflow stream, produced by the second  
7 IPS 4, was fed to the third mixer 6. This second underflow  
8 stream was quite lean in bitumen - more particularly, it was  
9 depleted in bitumen relative to the first underflow stream.

10          In the third mixer 6, the second underflow stream  
11 was mixed with fresh pure naphtha. The mixture was fed to the  
12 inlet of the third IPS 6 and underwent separation therein.  
13 The overflow stream from the third IPS 6 was recycled to the  
14 second mixer 3, as previously stated. The underflow stream,  
15 virtually free of bitumen, was discarded as tails.

16          The stream compositions and separation results are  
17 set forth in Table II.

1293465

TABLE II

## COMPOSITION (% BY WT.)

STREAM	BITUMEN	WATER	SOLIDS	NAPHTHA	RATE kg/min
Combined froth feed	57.3	34.2	8.5	-	1.96
First recycled overflow (from 2nd. IPS)	19.7	14.1	1.9	63.4	1.59
Overflow product (from 1st IPS)	55.7	4.7	0.7	39.0	2.02
1st IPS underflow	20.7	52.7	12.1	14.5	1.52
Second recycled overflow (from 3rd IPS)	2.80	53.8	8.3	35.1	3.01
2nd IPS underflow	2.9	13.7	74.6	9.3	2.95
Fresh diluent				99.5	0.81
3rd IPS underflow	0.20	77.3	20.3	2.4	0.75

SUPPLEMENTARY DISCLOSURE

This supplementary disclosure relates to a variation of the circuit described in the principal disclosure.

It can be advantageous to operate the separation process at elevated temperature because the viscosity of the hydrocarbon is thereby reduced. This allows the solid particles to settle more rapidly. In addition, at higher temperature the water droplets coalesce more readily, which facilitates their separation from the hydrocarbon. A high purity product is thereby produced at lower residence time, with the consequence that the capacity of the equipment is, in effect, increased.

At such higher temperatures, fractions of the diluent can approach or exceed their atmospheric boiling point. To prevent flashing of the diluent, and to contain the pressures generated, it is necessary to surround the functioning units of the equipment with pressure-retaining housings.

This may be effected in conventional fashion by closing in the components of the circuit, as indicated diagrammatically in Figure 3, and operating the process at elevated temperature and pressure.



1293465

1 THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE  
2 PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

3 1. A process for purifying bitumen froth from the hot  
4 water process for extracting bitumen from oil sand, said froth  
5 comprising bitumen, water and solids, said process being carried  
6 out in a circuit comprising first, second and third inclined  
7 plate settlers and first, second and third mixers, each settler  
8 having an inlet, an overflow outlet and an underflow outlet, each  
9 mixer having an inlet and outlet, each mixer being positioned  
10 before the corresponding settler, the outlet of each mixer being  
11 connected with the inlet of the immediately downstream settler,  
12 the inlet of the first mixer being connected with a source of  
13 bitumen froth, the inlet of the second mixer being connected with  
14 the underflow outlet of the first settler, the inlet of the third  
15 mixer being connected with the underflow outlet of the second  
16 settler, the overflow outlet of the first settler providing the  
17 diluted bitumen product from the circuit, the overflow outlet of  
18 the second settler being connected with the first mixer, the  
19 overflow outlet of the third settler being connected with the  
20 second mixer, the third mixer being connected with a source of  
21 light hydrocarbon diluent, said process comprising:  
22 mixing the bitumen froth in the first mixer with a  
23 first recycled overflow stream from the second settler, said  
24 overflow stream being depleted in bitumen and enriched in diluent  
25 relative to the froth;  
26 treating the mixture produced from the first mixer in  
27 the first settler to produce a first product overflow stream  
28 which is sufficiently enriched in bitumen relative to the froth  
29 to be of upgrading quality and a first underflow stream which is  
30 depleted in bitumen relative to the froth;

1293465

SECTION 8 CORRECTION  
SEE CERTIFICATE  
CORRECTION - ARTICLE 8  
VCM CERTIFICATE

1 depleted in bitumen and enriched in diluent relative to the first  
2 overflow stream;

3 treating the mixture produced from the second mixer in  
4 the second settler to produce the first recycled overflow stream  
5 and a second underflow stream which is depleted in bitumen  
6 relative to the first underflow stream;

7 mixing the second underflow stream from the second  
8 settler in the third mixer with a stream of light hydrocarbon  
9 diluent from said source of light hydrocarbon diluent;

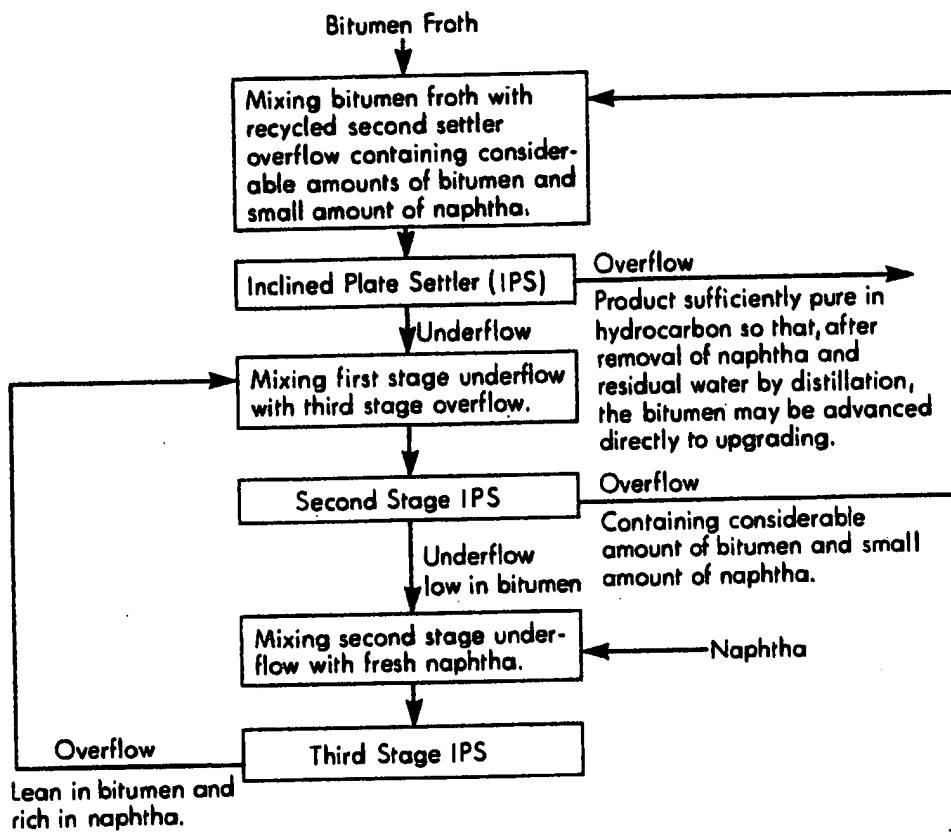
10 treating the mixture produced from the third mixer in  
11 the third settler to produce the second recycled overflow stream  
12 and a third underflow stream which is depleted in bitumen  
13 relative to the second underflow stream.

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1 CLAIM SUPPORTED BY THE SUPPLEMENTARY DISCLOSURE

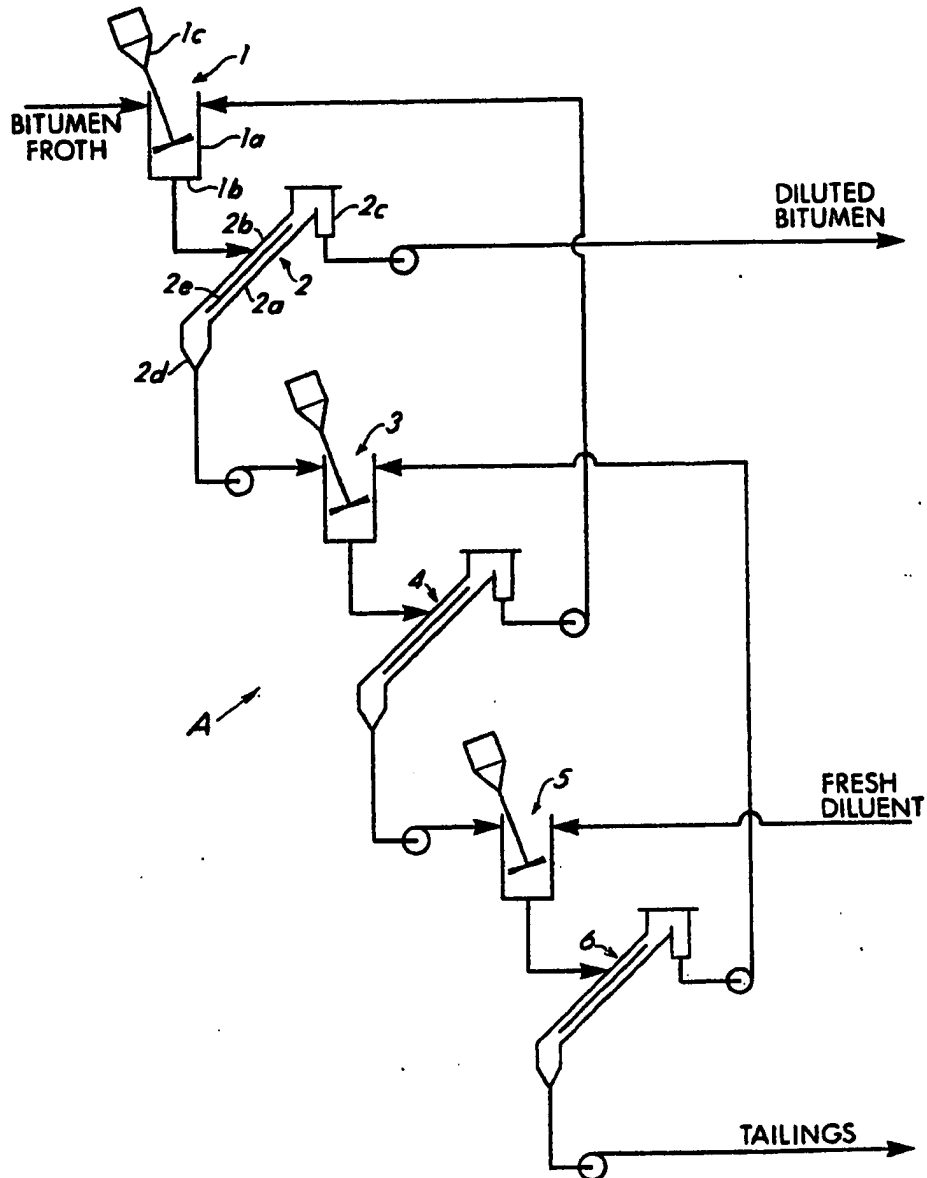
2           2. The process as set forth in claim 1 wherein:  
3           the process is conducted at elevated temperature and  
4           pressure and the circuit is pressure-retaining.



Fig. 1.

Patent agent:

*E. P. Johnson*

Fig. 2.

Patent agent:

*E. P. Johnson*

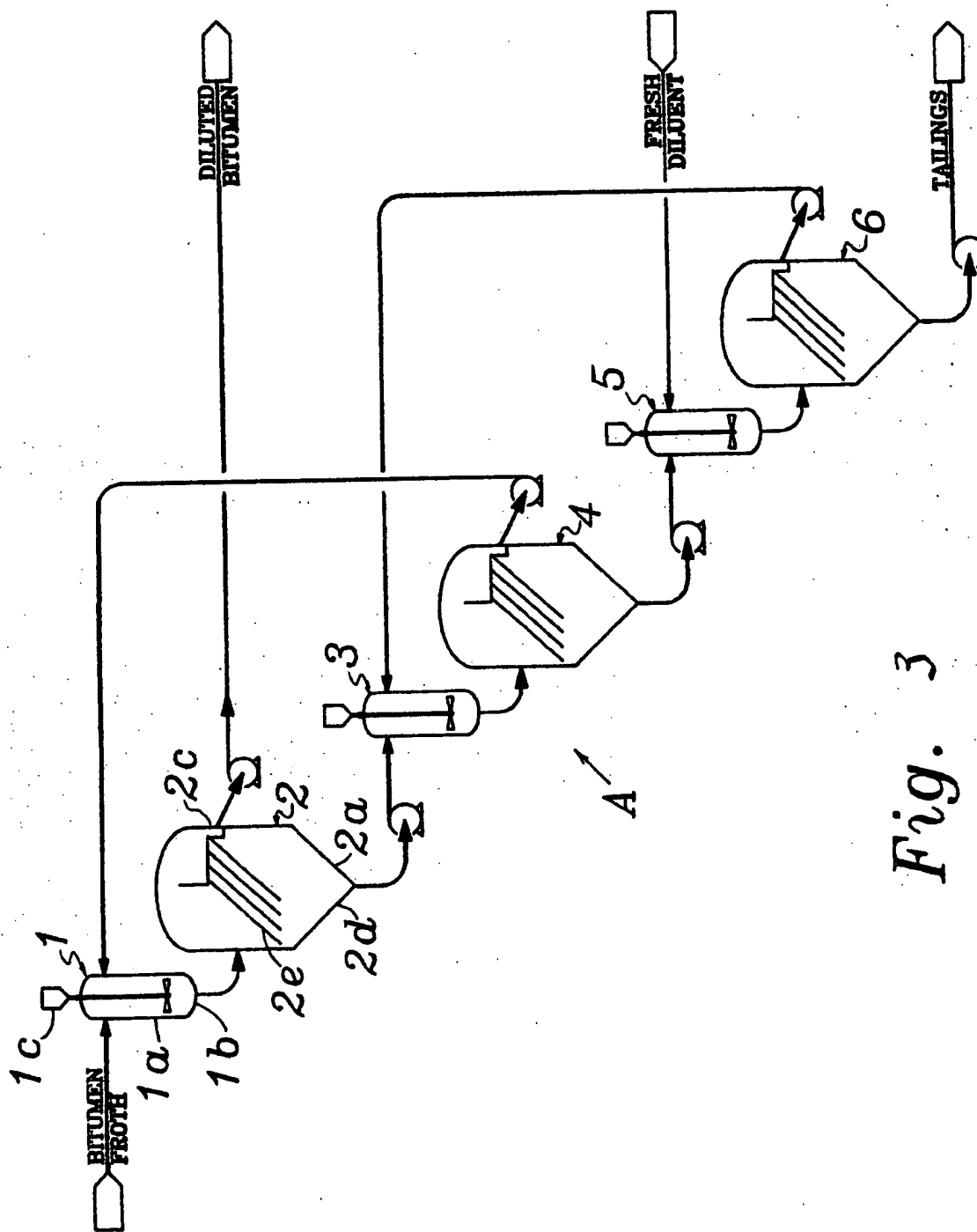


Fig. 3